DISCOVERY

57(311), November, 2021

Investigation of Cassava Starch Characteristics for Adhesive and Integrity of Preservative Methods

Ukpaka Chukwuemeka Peter✉, Gladys Nneoma Charles-Orji, Ikenyiri Patience Nna

To Cite:
Ukpaka CP, Gladys NCO, Ikenyiri PN. Investigation of cassava starch characteristics for adhesive and integrity of preservative methods. *Discovery*, 2021, 57(311), 732-748

Author Affiliation:
Department of Chemical/Petrochemical Engineering, Rivers State University Port Harcourt, PMB 5080, Rivers State, Nigeria

✉**Corresponding author:**
Department of Chemical/Petrochemical Engineering, Rivers State University Port Harcourt, PMB 5080, Rivers State, Nigeria
E-mail:chukwuemeka24@yahoo.com

Peer-Review History
Received: 29 August 2021
Reviewed & Revised: 30/August/2021 to 06/October/2021
Accepted: 09 October 2021
Published: November 2021

Peer-Review Model
External peer-review was done through double-blind method.

DISCOVERY
SCIENTIFIC SOCIETY

ABSTRACT

The research focussed on the need to preserve starch for the manufacture of adhesives particularly for regions were sunlight or drying facility is either not readily available or very uneconomical. Two key parameters were investigated during preservation, the viscosity and the microbial formation. The method involved the local extraction of two (2) starch species, two (2) each from primary starch sources of Cassava (TMS82/00058 and TMS 30001). Sulphuric acid (H_2SO_4) and Sodium Chloride (NaCl) were used as the acidic and alkaline preservatives respectively. The various starch samples were preserved in different concentrations of 0.1M, 0.3M, 0.5M, 0.7M, 0.9M, 1.0M for both preservatives and for a period of three (3) months. Starch samples in distilled water were used as control samples for all the two (2) starch species to check the effectiveness, degradation or enhancement of the acidic and alkaline preservatives. The viscosity of the preserved starch samples was measured at intervals of one (1) month throughout the period of the three (3) months. The viscosities were compared with those of the control sample. It was observed that the starch viscosities varied with the starch specie, for the control samples, with those samples in acidic and alkaline medium for the initial measurement. The samples were investigated for microbial growth over the duration of preservation and result showed that all control samples had microbial formation over the preservation periods. It was also significantly observed that the viscosity of the starch is affected by the presence of the preservatives at different levels of concentrations. Another significant observation was the variation of viscosity over preservation period for the same concentration of preservative for the starch species. The preservation was significantly effective for the 1.0M concentration of H_2SO_4 for all starch species since there was no microbe formation. For the cassava starch CV1, specie designated as when preserved in 1.0M H_2SO_4 concentration, however the alkaline preservation had reduction in viscosity over time. 5th order polynomial was used to fit the viscosity data for the prediction of viscosity from preservative concentration over time. The R^2 values for the predictive models were in the range of 0.98-1.0 indicating a high goodness of fit. The error estimation between the predicted and measured value were below 3.0%. Significantly, based on viscosity enhancement and non-microbial formations, majority of the investigated starch species can be preserved over a period of three

ISSN 2278–5469 EISSN 2278–5450 I OPEN ACCESS

months before use for adhesive production with 1.0M concentration of acidic (H_2SO_4) and alkaline (NaCl) preservatives.

Key words: Investigation, cassava starch, characteristics, adhesive, integrity, preservative methods

1. INTRODUCTION

As there continues a rise in environmental concerns about the industrial wastes generated from petroleum products and the potential consequences from most industrial activities, the use of natural, biodegradable, and renewable materials and substances has become more attractive. Starch is one of the most promising raw materials to produce biodegradable adhesives. Adhesives industries have grown and demand for starch adhesives has increased drastically, which has led developing countries like Nigeria to depend on locally sourced starchy materials for adhesive production [1-4]. But there is major drawback in the use of starch. This is the stability of the product over time. Starch usually degrades and discolour due to microbial attack, resulting to poor quality of starch [5].

Local farmers are discouraged from producing starch due to lack of equipment such as dryers for proper preservation of starch. So, they end up washing away starch residue that is produced during cassava processing [6]. Therefore, there is need to investigate the effect of preservatives on starch paste as a measure to determine the best and cheapest method to preserve starch paste for a long time and still retain its properties for production of goods like adhesive [7-10].

The aim of this study is to investigate the effect of acidic and alkaline preservative mediums on starch paste obtained from varieties of cassava for the production of adhesives. The aim of this study will be achieved or actualized by the following objectives which are to: Determine the mode of extraction of starch from Cassava, analysis and packaging of the starch produced, preparation and application of different concentrations of the preservative agents on the produced starch, investigate the pH level at which the starch is being preserved starch to determine microbial survival rate, viscosity analyses for 3months of preservation period at 1month interval as the viscosity is the key parameter to determine good quality starch and determine which starch variety would withstand the preservative agents when used to produce adhesive.

This study covers the production of starch from various species of cassava, potatoes and corn, the preservation of the starch pastes with acidic and alkaline mediums preservative for production of stable adhesive in future, monitoring the pH of preserved starch, and comparing the effect of the preservative concentration on the viscosity.

As the major drawback in the use of starch for adhesive and other products, is the stability of the product over time, this research will help to achieve the prolonged preservation of starch while still maintaining the desired properties of starch for a stable adhesive. This study will benefit local farmers especially cassava processors as it tends to improve their knowledge on easy preservation of starch paste and encourage them to go into adhesive production.

The significance of the study is attributed to the importance of viscosity as key property for determining quality of starch used for adhesive. The viscosity of the preserved starch was determined to ensure satisfactory quality of starch. The monitoring of the pH of the preserved starch is significant to monitor the rate of microbial growth.

2. MATERIALS AND METHODS

The experiments were performed at the Unit Operations Laboratory of the Department of Chemical/Petrochemical Engineering, Rivers State University of Science and Technology, Port-Harcourt.

Materials

Plant Materials: The different varieties of starchy crops are gotten from the Rivers State Agricultural Development Program (ADP) research farm in Port Harcourt. Table 1 below below specify the starch sources and the varieties.

Table 1: Species of Starch Sources used for Investigation

s/no	Starch source	Species	
A	Tubers		
1	Cassava(10kg each)	CV1-TMS82/00058	CV2-TMS 30001

Instruments/Equipment used:1000g digital weighing balance, 50ml and250ml glass Pyrex Beakers, 50ml glass Pyrex measuring cylinder and 1000ml plastic Pyrex measuring cylinder, Spatula, Funnel, 360°C Pyrex Thermometer,30ml Pyrex Pycnometer, Pyrex

crucible, DHG-9140A Drying Oven,pH-618 RoHpHmeter with Mettler Toledo technical Buffer Solution, Gerhardt hot plate and NDJ-8S Digital Display Viscometer.

Chemical/Reagents: The two categories of preservative reagent used are the acidic and alkaline medium. For the acidic medium, Analar Concentrated sulphuric acid (H_2SO_4) was used and for the alkaline medium, Dangote table salt (Concentrated Sodium Chloride (NaCl)) was used. Distilled water (H_2O) was used as a control preservative to serve as a base for comparative analysis to determine the impacts of preservatives on starch species. Table 2 below specifies the preservative reagents used.

Table 2: Preservative Reagents

Acidic Preservative	Alkaline Preservative	Control
190ml Conc. H_2SO_4	205g Conc. NaCl	1000ml distilled H_2O (12portions)

Method of Experiment

This research has to do with chemical Extraction process. Extraction is the process of separating one or more components from a liquid mixture as the required component dissolves out with or without the aid of a solvent. The experimental procedure began with the extraction of the raw starch from the different species of starch sources. The exact processes are explained in detail below. Another vital aspect is the preparation of the reagents used as preservatives.

Extraction of Starch

For this study, starch was extracted from 2 different varieties each of starchy Tubers-Cassava. The basic unit operations involved in the extraction of starch from tuber crops are highlighted in Figure 1.

Starch Extraction from Tubers

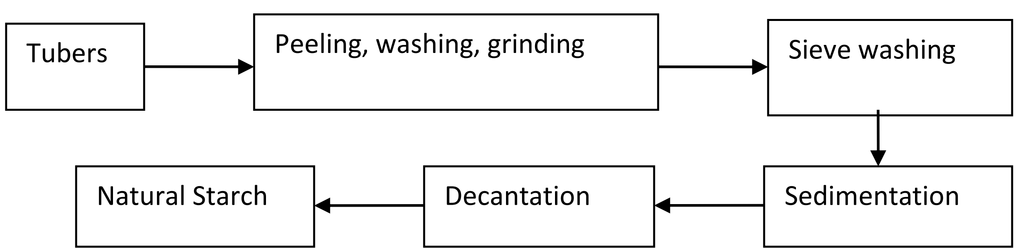

Figure 1: Flow Diagram for Starch Extraction from Tubers (Cassava).

Cassava Starch Extraction

The Cassava starches were extracted as described in the research conducted by Ukpaka et al [11]. The fresh tubers of cassava speciesTMS82/00058(CV_1) were washed with water to remove sand and dirt. The outer layers of the cassava tubers were peeled manually with a knife. The peeled cassava tubers were washed again with clean tap water to clean it from dirt. These were then grated with a grating machine. A large amount of water was added to the grated cassava and mixed thoroughly. The mixture was sieved using a cloth sieve and the filtrate was allowed for sedimentation for 6hours in a bucket, after which the raw starch settles at the bottom of the bucket and the water above the starch is decanted. The sediment starch was washed two times with clean water to obtain a pure white starch. The starch was collected and put in a cloth bag and squeezed to remove extra water. The starch was then weighed and put in covered plates to be mixed with the preservatives. This process is repeated for all the tubers; cassava species TMS 30001(CV_2).

Preparation of Reagents

The procedure for calculating and preparation of the reagents described by Ukpaka,et al. [11] was adapted. The reagents were chosen paying attention to what the preserved starch will be used for (whether edible or contact with the skin) as well as not influencing the properties of the preserved starch and the room temperature at which the starch would be preserved. The reagents were prepared in different concentrations for the acidic medium and the alkaline medium. This is to determine what concentration will best preserve the starch.

ISSN 2278–5469 EISSN 2278–5450 I OPEN ACCESS

Calculation for Portions of H_2SO_4 used to prepare the Acidic Preservatives.

The molarity and dilution equations were used to calculate the amount of H_2SO_4used to prepare the required preservative concentration. Molarity (M), also known as the molar concentration of a solution is the amount of a substance in a certain volume of solution. Molarity is defined as the moles of a solute per litres of a solution (moles of solute / litres of solution).

Also given is the Assay also known as purity. This is a measure of the composition and quality of a chemical substance. Molarity can then be calculated giving the assay with the formula below.

$$M = Assay * \rho * W \quad \textbf{(1)}$$

Where M = Molarity

Assay is the average value of the actual concentration of the undiluted acid as seen on the bottle. ρ = density and W = relative molecular mass.

Preparation of various Portions of Sulphuric acid (H_2SO_4)

From the calculations shown in appendix for **H_2SO_4** the following were obtained.

5.4ml of concentrated sulphuric acid was measured into a volumetric flask containing 1000ml of distilled water to form 0.1M of (H_2SO_4) acid.

16.3mls of (H_2SO_4) was measured into 1000ml of distilled H_2O to form 0.3M of (H_2SO_4).

27.1mls of (H_2SO_4) was measured to 1000ml of distilled $H_2O$to form 0.5M of (H_2SO_4).

37.97ml of H_2SO_4 was mixed with 1000ml of distilled water to form 0.7M of (H_2SO_4).

48.82ml of H_2SO_4 was mixed with 1000ml of distilled water to form 0.9M of (H_2SO_4).

54ml of H_2SO_4 was mixed with 1000ml of distilled water to form 1.0M of (H_2SO_4).

Calculation for Portions of NaCl used to prepare the Alkaline Preservative

The molarity and Dilution equations are also used to determine the mass of NaCl needed to prepare the various concentrations of the alkaline preservative. The equation is given as:

$$n = CV \quad (2)$$

Converting moles to the mass needed we have;

$$m = nM \quad (3)$$

Where $\boldsymbol{n}$ is the number of moles, $\boldsymbol{m}$ is the mass of NaCl needed, $\boldsymbol{M}$ is the molar mass of NaCl

C, V is concentration and volume respectively

Preparation of Various Portions of NaCl Salt

From the calculations shown in appendix D for NaCl the following were obtained.

i. 5.8g of NaCl dissolved in 1000ml of distilled H_2O to form0.1M of NaCl.
ii. 17.5g of NaCl dissolved in 1000ml of distilled H_2O to form 0.3M of NaCl.
iii. 29.2g of NaCl dissolved in 1000ml of distilled H_2O to form 0.5M of NaCl.
iv. 40.9g of NaCl dissolved in 1000ml of distilled H_2O to form 0.7M of NaCl.
v. 52.6g of NaCl dissolved in 1000ml of distilled H_2O to form 0.9M of NaCl.
vi. 58.4g of NaCl dissolved in 1000ml of distilled H_2O to form 1.0M of NaCl.

Preservation of Starch

The starch was preserved in a ratio of 1:1 for the starch and the preservative reagents. 50g of CV_1-TMS82/00058 was weighed and put in different containers. 50mls of 0.1M of H_2SO_4 was measured and mixed properly with the starch to form a paste. This was labelled $CV1Ap_{0.1M.}$ This was repeated for other concentrations of the acidic medium and alkaline medium preservative agents. This was then repeated for other varieties of the starch. The containers were labelled according to the different starch produced and the different concentrations of acid medium preservative as (AP) and alkaline medium preservative medium as (SP). A total of 78 samples were collected for this research and labelled as follows:

ISSN 2278–5469 EISSN 2278–5450 I OPEN ACCESS

$CV1Ap_{0.1M}$, $CV1Ap_{0.3M}$, $CV1Ap_{0.5M}$, $CV1Ap_{0.7M}$, $CV1Ap_{0.9M}$, $CV1Ap_{1.0M}$ for CV1(TMS82/00058) preserved with AP(H_2SO_4).
$CV1Sp_{0.1M}$, $CV1Sp_{0.3M}$, $CV1Sp_{0.5M}$, $CV1Sp_{0.7M}$, $CV1Sp_{0.9M}$, $CV1Sp_{1.0M}$for CV1 (TMS82/00058) preserved with SP (NaCl).
$CV2Ap_{0.1M}$–$CV2Ap_{1.0M}$ for CV_2(TMS 30001) preserved with AP(H_2SO_4).
$CV2Sp_{0.1M}$ – $CV2Sp_{1.0M}$ for CV_2(TMS 30001) preserved with SP(NaCl).

3. RESULTS AND DISCUSSION

Effect of Starch Preservative on Viscosity, pH, and Density

The effect of the preservative types on the various starch species are explicitly presented and discussed in the forgoing sections. The properties investigated include the viscosity, the pH, the Density as well as the physical nature of the starch paste over the duration of preservation. The six varieties were treated with the two different types of diluted concentrations of the preservatives for duration of 3months. The viscosity and density properties were measured after intervals of 1month for a period of three months. As benchmark samples, the properties of the starch paste (starch: water ratio of 1:1, 50g) without Alkaline or Acidic preservatives were also measured for the different starch species for a period of 3 months at the interval of 1 month. These properties were compared with samples with preservatives to see the extent of decay or viscosity enhancement as well as the microbial formations over the period under investigation.

The results are presented in Table 3.

Table 3: Viscosity, Density and pH of the Benchmark Samples (Water Preservative)

			INITIAL		1MONTH		2MONTHS		3MONTHS	
Starch Type	**Starch and water (g); ratio (1:1) (1:1)**	**pH (-)**	**Viscosity (pa. s)**	**Density (g/cm^3)**	**Viscosity (pa. s)**	**Density (g/cm^3)**	**Viscosity (pa. s)**	**Density (g/cm^3)**	**Viscosity (pa. s)**	**Density (g/cm^3)**
CVI	50	5.9	315.9	1.386	315.7	1.364	341.8	1.345	367.7	1.365
CV2	50	6.1	340.3	1.365	328.3	1.353	358.3	1.352	377.6	1.352
PT1	50	5.2	425.5	1.327	374.7	1.349	339.15	1.322	252.8	1.343
PT2	50	5.4	397.5	1.07	327.6	1.267	322.6	1.093	246.5	1.152
CO1	50	5.5	170.7	1.357	164.9	1.366	171.8	1.324	217.8	1.352
CO2	50	4.3	158.2	1.266	161.5	1.287	172.4	1.284	196.3	1.276

Table 4: Identification and Characterization of Microbes Grown in Preserved Starch Paste

SAMPLE	MACROSCOPY	MICROSCOPY	IDENTIFIED ORGANISM
CV1AP0.1M	Same as above	Same as above	1.Alternaria Sp. 2.Aspergillus Niger.
CV1AP0.3M	White fluffy growth with yellow reverse.	Septate branding hyphae, with banana shape conidia same as above	1.Fusarium Sp. 2.Alternaria Sp.
CV1AP0.5M	Brown colony growth with white periphery and yellow reverse.	Septet hyphae, columnar conidia, head spores.	1. Aspergillus Tereus. 2. Aspergillus Niger. 3. Yeast Sp.
CV1SP0.9M	Same as above	Same as above	1. Aspergillus Niger
CV1AP1.0M	No growth	No growth	No growth
CV1Water	Milk colour growth.	Colloid cells.	1. Yeast Sp.

Table 5: Microbial Count in some of the Samples

Preserved Starch Sample	Colony Forming Units
CV1AP0.1M	$0.4*10^5$
CV1AP0.3M	$0.02*10^5$
CV1AP0.5M	$0.4*10^5$
CV1SP0.9M	$0.7*10^5$
CV1 Water	$25.9*10^5$

ISSN 2278–5469 EISSN 2278–5450 I OPEN ACCESS

CV1AP1.0M	No growth
CV2AP1.0M	No growth

Effect of Preservative Agents on CV1 (TMS82/00058)

Table 6 and 7 shows the result for the cassava starch group CV1 (TMS82/00058) when preserved using acidic (H_2SO_4) and alkaline (NaCl) preservatives respectively. The variation in viscosity as shown in Figure 2 and 3 with the pH for the various concentrations are captured in Figures 4 and 5 for acidic and alkaline preservatives respectively.

Table 6: CV1 (TMS82/00058) Preserved in Acidic Medium (H_2SO_4)

Acidic Preservative conc. (M)	Mass of CV1 Starch (g)	pH (-)	INITIAL		1MONTH		2MONTHS		3MONTHS	
			Viscosity (pa. s)	Density (g/cm^3)	Viscosity (pa. s)	Density (g/cm^3)	Viscosity (pa. s)	Density (g/cm^3)	Viscosity (pa. s)	Density (g/cm^3)
0.1	50	3.5	179.3	1.366	196.8	1.347	230.6	1.352	256.5	1.348
0.3	50	3.3	185.4	1.427	199.9	1.400	240.0	1.408	294.6	1.399
0.5	50	2.5	222.7	1.328	231.6	1.406	249.9	1.415	277.1	1.400
0.7	50	2.3	260.0	1.449	236.5	1.342	259.9	1.432	259.6	1.220
0.9	50	2.2	445.9	1.333	407.0	1.324	399.4	1.315	422.6	1.311
1.0	50	2.1	631.8	1.321	571.9	1.256	538.9	1.387	585.2	1.245

Table 7: CV1 (TMS82/00058) Preserved in Alkaline Medium (NaCl)

Acidic Preservative conc. (M)	Mass of CV2 Starch (g)	pH (-)	INITIAL		1MONTH		2MONTHS		3MONTHS	
			Viscosity (pa. s)	Density (g/cm^3)	Viscosity (pa. s)	Density (g/cm^3)	Viscosity (pa. s)	Density (g/cm^3)	Viscosity (pa. s)	Density (g/cm^3)
0.1	50	3.9	195.8	1.348	201.8	1.286	224.68	1.298	235.82	1.287
0.3	50	3.4	240.7	1.427	224.6	1.293	237.24	1.372	265.34	1.292
0.5	50	2.9	243.8	1.320	237.1	1.339	240.12	1.473	249.26	1.333
0.7	50	2.6	260.3	1.360	233.6	1.512	253.21	1.365	268.78	1.375
0.9	50	2.4	407.5	1.425	381.3	1.396	384.46	1.372	415.26	1.382
1.0	50	2.0	652.9	1.388	591.1	1.296	557.21	1.275	553.01	1.285

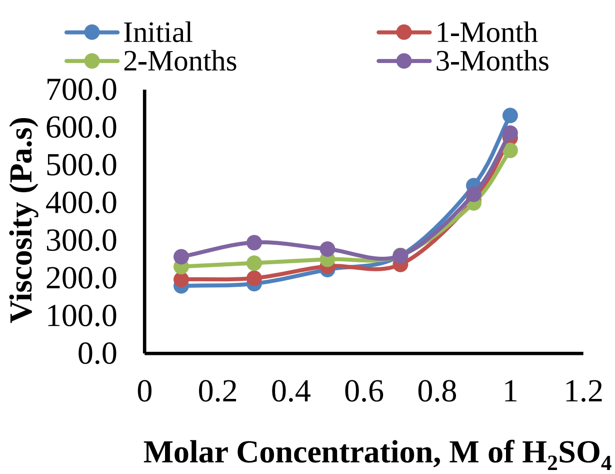

Figure 2: Graph of Viscosity vs Molar Concentration for H_2SO_4 Preservative for CV1.

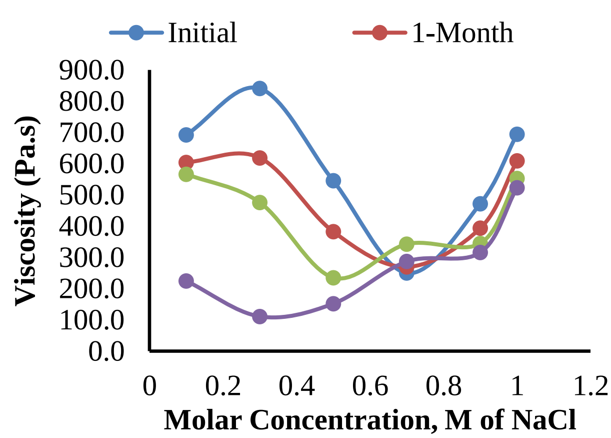

Figure 3: Graph of Viscosity vs Molar Concentration of NaCl Preservative for CV1.

ISSN 2278–5469 EISSN 2278–5450 I OPEN ACCESS

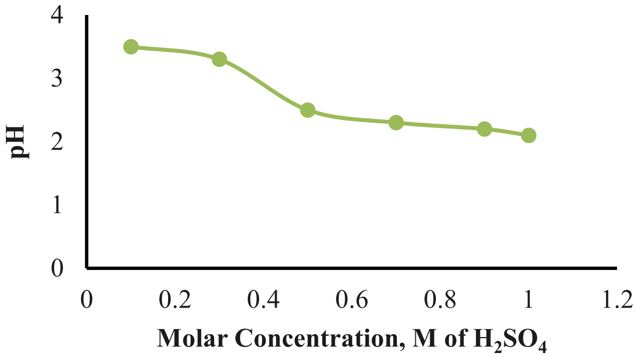

Figure 4: Graph of pH vs Molar Concentration of H_2SO_4 Preservative for CV1.

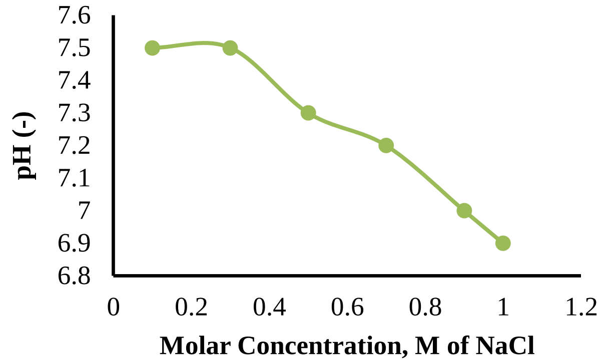

Figure 5: Graph of pH vs Molar Concentration of NaCl Preservative for CV1.

When preserved in acidic solution, the viscosities for all the months are seen to averagely vary in the upward direction with increasing concentration of the solution. The pH however shows a decrease with increasing concentration showing it is acidic as it decreases downwards away from the control value of 5.9 to 2,1 for a 1.0M of acidic solution. A comparative analysis with the initial viscosity showed that the viscosity reduced from 315. 9Pa.sto179.3 (43.24% drop) on preserving with a concentration of 0.1M and this is in line with observations from (Emblem *et al.* 2012) and increased thereafter peaking at 631.8Pa.sindicative of a 50% rise at a concentration of 1.0M which is ten(10) times higher than 0.1M. During the second month of preservation, the control viscosity increased to 341.8Pa.s showing a gain in 25.9Pa.s (7.36%). This is due to the increase in the coagulation resulting from the time of preservation. Similar behaviour was observed at the 3rd month where the control viscosity of 367.7Pa.s reduced to 256.5Pa.s at0.1M concentration and peaked at 585.2Pa.s with the 1.0M concentration. Uniquely for all the months of preservation, the control viscosities though initially diminishing as the concentration increased were restored from 0.7M.and peaked at 1.0M. Comparing the viscosity preservation and the microbial growth rate, although the viscosity values are generally good from 0.7M, there were microbial formations on the samples for all cases except for the concentration of 1.0M of preservative concentration. It is thus pertinent to conclude that the cassava starch specie CV1 is best preserved both in viscosity and usable form when the concentration of the acidic preservative (H_2SO4) is 1.0M for at least a minimum of 3months before usage.

The same conclusion is adduced for the alkaline preservative as well looking at the preserved values of the viscosities of 694.2, 604.4, 552.6 and 523.45Pa.s for initial condition, months 1, 2, and 3 respectively at alkaline concentration of 1.0M as no microbial growth was observed.

Effect of Preservative Agents on CV2 (TMS 30001)

The results obtained for the cassava starch specie CV2 (TMS 30001) are shown in Tables 8 and 9, and the graphical representations of the viscosity and the pH for the preservative concentrations used are captured in figures 4.6 to 4.9. From the observed trends, the behaviour of the CV1 is very similar to that of CV2 because of the common primary source which is cassava.

Table 8: CV2 (TMS 30001) Preserved in Acidic Medium (H_2SO_4)

Acidic Preservative conc. (M)	Mass of CV2 Starch (g)	pH (-)	INITIAL		1MONTH		2MONTHS		3MONTHS	
			Viscosity (pa. s)	Density (g/cm^3)	Viscosity (pa. s)	Density (g/cm^3)	Viscosity (pa. s)	Density (g/cm^3)	Viscosity (pa. s)	Density (g/cm^3)
0.1	50	3.9	195.8	1.348	201.8	1.286	224.68	1.298	235.82	1.287
0.3	50	3.4	240.7	1.427	224.6	1.293	237.24	1.372	265.34	1.292
0.5	50	2.9	243.8	1.320	237.1	1.339	240.12	1.473	249.26	1.333
0.7	50	2.6	260.3	1.360	233.6	1.512	253.21	1.365	268.78	1.375
0.9	50	2.4	407.5	1.425	381.3	1.396	384.46	1.372	415.26	1.382
1.0	50	2.0	652.9	1.388	591.1	1.296	557.21	1.275	553.01	1.285

ISSN 2278–5469 EISSN 2278–5450 I OPEN ACCESS

Table 9: CV2 (TMS 30001) Preserved in Alkaline Medium (NaCl)

Alkaline Preservative conc. (M)	Mass of CV2 Starch (g)	pH (-)	INITIAL Viscosity (pa. s)	INITIAL Density (g/cm^3)	1MONTH Viscosity (pa. s)	1MONTH Density (g/cm^3)	2MONTHS Viscosity (pa. s)	2MONTHS Density (g/cm^3)	3MONTHS Viscosity (pa. s)	3MONTHS Density (g/cm^3)
0.1	50	7.2	652.84	1.319	593.6	1.315	583.84	1.325	246.3	1.316
0.3	50	7.1	820.27	1.338	611.9	1.325	481.72	1.338	151.78	1.324
0.5	50	7.0	542.83	1.361	442.9	1.334	361.01	1.358	187.5	1.328
0.7	50	7.1	251.31	1.318	288.8	1.479	383.4	1.309	298.21	1.345
0.9	50	6.9	467.53	1.323	414.1	1.378	392.5	1.316	330.51	1.364
1.0	50	6.9	752.45	1.358	670.5	1.381	620.1	1.328	552.47	1.369

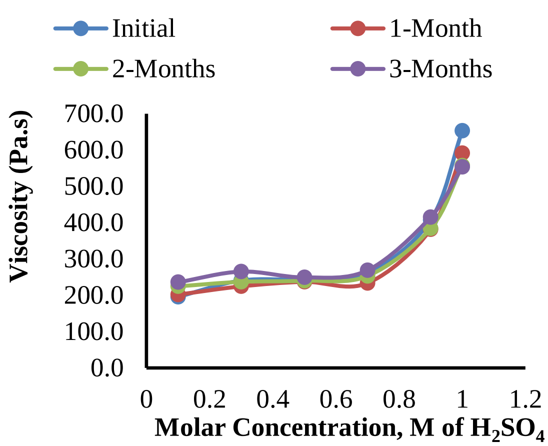

Figure 6: Graph of Viscosity vs Molar Concentration of H_2SO_4 Preservative for CV2.

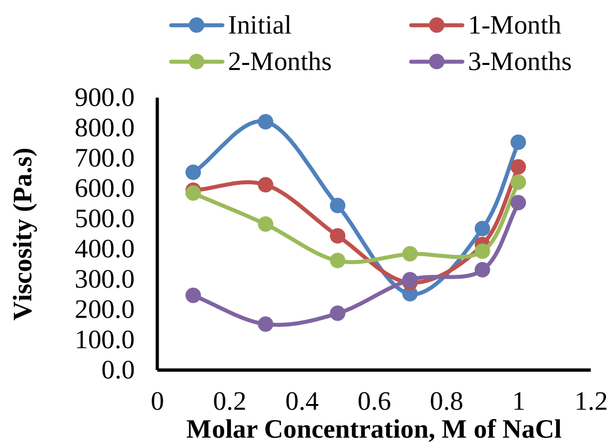

Figure 7: Graph of Viscosity vs Molar Concentration of NaCl Preservative for CV2.

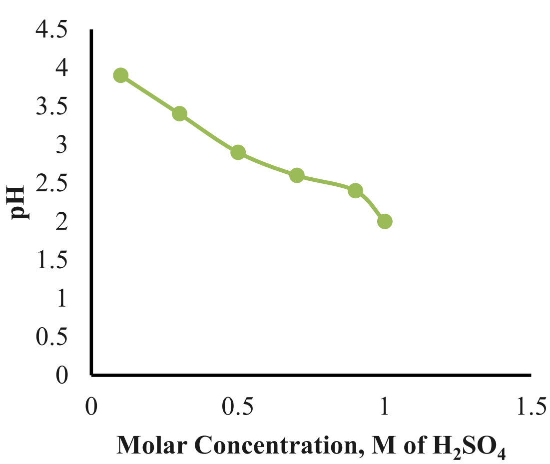

Figure 8: Graph of pH vs Molar Concentration of H_2SO_4 Preservative for CV2.

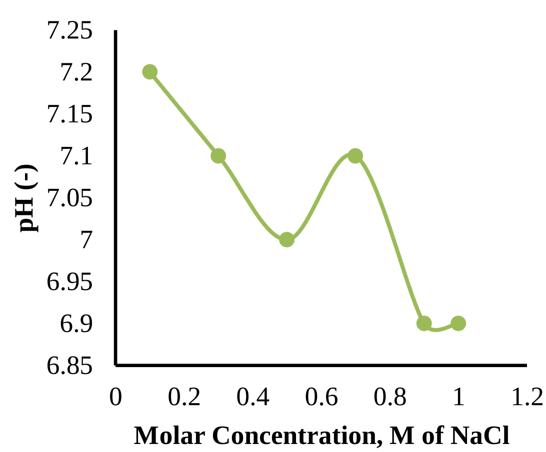

Figure 9: Graph of pH vs Molar Concentration of NaCl Preservative for CV2.

With CV2 preserved in acidic solution, the viscosity for all the months increased with concentration of the solution. The pH however shows a decrease with increasing concentration, decreasing downwards away from the control value of 6.1 to 2.0 for a 1.0 M of acidic solution. Comparing the viscosity trend in months with that of the control sample, at the start of the preservation, the viscosity reduced by 42.46%, i.e. from 340.3Pa.s to 195.8Pa.sat a concentration of 0.1M and increased thereafter to a maximum value of 652.9Pa.s indicative of a 47.88% rise at a concentration of 1.0M. Within 1 month of preservation in 1.0M, the viscosity reduced by 61.8Pa.s or 9.47% because of a weakening bond between the wet starch viscous layers after 2months of preservation, the control

ISSN 2278–5469 EISSN 2278–5450 I OPEN ACCESS

viscosity was observed to be 18Pa.shigher which translates to a 52.89% gain. Similar behaviour was observed at the 3rd month where the control viscosity of 377.6Pa.s reduced to 268.78Pa.s at 0.7M concentration and then increased to 553.01Pa.s when preserved in 1.0M concentration. Generally, throughout the 3 months of preservation studied, the control viscosities diminished initially as the concentration increased and started rising from 0.7M and peaked at 1.0M. Again, comparing the preservation of viscosity and the usability based on microbial growth rate, the viscosity values are generally adjudged good from 0.7M. However, there were microbial formations on the samples for all cases except for the concentration of 1.0M of preservative concentration. It is then important to suggest that the wet cassava starch specie CV2 can be preserved for a period of 3months both in viscosity and usable form when the concentration of the acidic preservative (H_2SO_4) is 1.0M.

The same conclusion is adduced for the alkaline preservative as well looking at the preserved values of the viscosities of 752.45, 670.5, 620.1 and 552.47Pa.s for initial condition, one, two and three months respectively at alkaline concentration of 1.0M since no microbial growth was observed as well. At this concentration, the viscosities in alkaline preservative are richer compared to those of acidic concentration by 15.25%, 11.84% and 11.29% for initial or start of preservation, after month 1 and month 2 respectively. However, during the 3rd month of preservation the values are approximately the same showing a small difference of -0.098%.

4. CONCLUSION

The research focussed on the need to preserve starch for the purpose of using it for the manufacture of adhesives particularly for regions where sunlight or drying facility is either not readily available or very expensive. Amidst other starch properties, two key parameters were investigated to determine the quality of starch before and after preservation. These include the viscosity; which is the key parameter to determine the quality of starch and the microbial analysis; to determine the level of microbial formation after preservation. The method involved the use of two different starch species from each of the primary sources which are different species of cassava. Two preservative media were used; the acidic medium is a sulphuric acid (H_2SO_4) and the alkaline medium is sodium chloride (NaCl) with different concentrations. This is to determine what concentration will best preserve the starch. The concentrations are 0.1M, 0.3M, 0.5M, 0.7M, 0.9M, 1.0M concentrations of H_2SO_4 and NaCl solutions. Distilled water without acidic or alkaline preservative was used as control or benchmark in order to check the effectiveness, degradation or enhancement of the viscosity of the preserved starch. The viscosity is measured for all the different starch samples for a period of 3 months at the interval of 1 month and compared using the control sample as benchmark. Conclusively, the following observations were recorded thus:

1. The viscosity trend showed variation amongst the different starch types. This could be due to the difference in molecular weight of the amylose portion and the ratio of amylose to amylopectin as reported by Anahita, [12]. As seen for the cassava starch (CV1 and CV2) maintained a viscosity that appeared enhanced over time ranging from 315.9 Pa.s (initial viscosity) to 367.7 Pa.s (@ month 3). However, the microbial analysis showed that the samples were not significantly preserved due to microbial formations found on them after the period of preservation, which is similar to the result obtained by Lee [13].This moved on to see how the viscosities were affected at the addition of the acidic and alkaline preservatives.
2. A 43.24% drop in viscosity was recorded at the addition of 0.1Mconcentration of the acidic preservative to the cassava starch CV1 (TMS82/00058), compared with the benchmark CV1 sample (from315.9Pa.s to 179.3Pa.s).The pH however showed a decline with concentration value of 5.9 to 2.1 for 1.0M concentration, which shows it is acidic. Uniquely for all the months of preservation, the viscosities showed initial diminishing which was restored as concentration increased to 0.7M and peaked at 631.8Pa.s for1.0M concentration showing 50%. Increase. Comparing the viscosity preservation and the microbial growth rate, although the viscosity values are generally good from 0.7M, there were microbial formations on the samples for all cases except for the 1.0M concentration of the acidic preservative. It is thus pertinent to conclude that the cassava starch specie CV1 is best preserved both in viscosity and usable form with1.0M concentration of the acidic preservative (H_2SO4) for at least a minimum of 3months before usage. The same conclusion is drawn for the alkaline preservative with good viscosities ranging from 694.2, 604.4, 552.6 to 523.45Pa.s for initial condition, months 1, 2, and 3 respectively at alkaline concentration of 1.0M with no microbial growth observed. The behaviour of the cassava starch CV1 (TMS82/00058) preserved in acidic concentrations are similar to those observed for the cassava starch CV2 (TMS 30001). This could be because they of the common primary starch source which is cassava.
3. Mathematical Models for Predicting Viscosity and Concentration for Starch Preservation were developed using the regression analysis with a polynomial model. The R^2 value was monitored for best fit which led to the predominant use of the 5th order polynomial.The R^2 values for the models range from 0.98 to 1, indicating the goodness of the polynomial fit for the data used. The models are validated simply from the measured values of the data. These models are also useful for the determination of

ISSN 2278–5469 EISSN 2278–5450 I OPEN ACCESS

the viscosity of the sample for any expected duration by linearly interpolating in between periods as detail description is demonstrated in the appendix.

Appendix

Starch Sources

(a) Cassava (TMS82/00058)

(b) (TMS30001)

Starch Extraction Process

(a) Grinding of Peeled Cassava

(b) Ground Cassava in abag

(c) Sieve Washing

(d) Decantation

ISSN 2278–5469 EISSN 2278–5450 I OPEN ACCESS

(e) Natural White Starch

(f) Sedimentation

Appendix C

Extracted Starch Sample Preparation and Treatment

(a)Researcher in the Lab Mixing Samples

(b)Packed Samples (Starch +Preservation)

(c)Preserved Starch Samples ready for Measurement

(d)Gelatinized Starch in Beakers

(e) Insufficiently Dried Starch attacked by Moulds

(f) Sample under a Viscometer

Manual Calculations for Preparation of Reagents

D1: Calculation for Portions of H_2SO_4 used to prepare the Acidic Preservative.

The equation below was used to determine the volume of H_2SO_4 needed to prepare the preservative. Equation (D.2) below demonstrates the dilution equation

$$C_1 * V_1 = C_2 * V_2 \quad \textbf{(D.1)}$$

So that $V_1 = \frac{C_2 * V_2}{C_1}$ **(D.2)**

ISSN 2278–5469 EISSN 2278–5450 I OPEN ACCESS

Where,
C_1 = concentration of the concentrated acid (M)
V_1 = Volume of H_2SO_4 needed to prepare the diluted solution.
C_2= Concentration of the diluted solution (0.1M, 0.3M, 0.5M, 0.7M, 0.9M and 1.0M respectively).
V_2= Total Volume of the solution (1000ml)

Given the following **details from the periodic table**

$$\text{Molecular mass of } \mathrm{H_2SO_4} = 98\mathrm{g}$$
$$\text{Desity of } \mathrm{H_2SO_4} = 1843.7\mathrm{g/L}$$
$$\text{Specific Gravity} = 1.8437$$
$$\text{Assay} = 98\%$$

Substituting these values into equation (3.1), the molar concentration of H_2SO_4 will be as follows:

$$C_1 = \frac{98g}{100g} * \frac{1843.7g}{1solution} * \frac{1m0lH_2SO_4..}{98.0g}$$
$$\mathrm{C_1} = 18.437\text{mole/liter}$$

18.437mole/litre is the molar concentration of the undiluted H_2SO_4.
Substituting values into equation (D.2), we have

For 0.1M

$$V_1 = \frac{0.1M * 1000ml}{18.437}$$
$$V_1 = 5.4ml$$

5.4ml of H_2SO_4 is needed to prepare 0.1M of H_2SO_4 preservative

For 0.3M

$$V_1 = \frac{0.3M * 1000ml}{18.437}$$
$$V_1 = 16.3ml$$

For 0.5M

$$V_1 = \frac{0.5M * 1000ml}{18.437}$$
$$V_1 = 27.1ml$$

For 0.7M

$$V_1 = \frac{0.7M * 1000ml}{18.437}$$
$$V_1 = 37.97ml$$

For 0.9M

$$V_1 = \frac{0.9M * 1000ml}{18.437}$$
$$V_1 = 48.82ml$$

For 1.0M

$$V_1 = \frac{1.0M * 1000ml}{18.437}$$
$$V_1 = 54.0ml$$

D2: Calculation for Portions of NaCl used to prepare the Alkaline Preservative

The equation below was used to determine the amount of NaCl needed to prepare the preservative.

$$n = CV \tag{D.3}$$

ISSN 2278–5469 EISSN 2278–5450 I OPEN ACCESS

Converting moles to the mass needed we have;

$m = nM$ (D.4)

Where $\boldsymbol{n}$ is the number of moles.

$\boldsymbol{m}$ is the mass of NaCl needed

$\boldsymbol{M}$ is the molar mass of NaCl

$\boldsymbol{C}, \boldsymbol{V}$ is concentration and volume respectively.

The following details are got from the periodic table

Molecular mass of NaCl = 58.5g

Desity of NaCl = 2163g/L

Specific Gravity = 2.163

Assay = 46%

For 0.1M

$\mathrm{n} = 0.1\frac{\mathrm{mol}}{\mathrm{litre}} \times 1\mathrm{litre}$

= 0.1moles

Substituting into equation (D.4)

$m = 0.1 \times 58.44$

= 5.844g NaCl is needed.

For 0.3M

Similarly, using equation (D.3) and (D.4)

$\mathrm{n} = 0.3\frac{\mathrm{mol}}{\mathrm{litre}} \times 1\mathrm{litre}$

= 0.3 moles

Substituting into equation (D.4)

$m = 0.3 \times 58.44$

= 17.532g NaCl is needed.

For 0.5M

Again, using equation (D.3) and (D.4)

$n = 0.5\frac{mol}{litre} \times 1litre$

= 0.5 moles

Substituting n into equation (D.4)

$m = 0.5 \times 58.44$

= 29.22g NaCl is needed.

For 0.7M

Using equation (D.3) and D.4)

$n = 0.7\frac{mol}{litre} \times 1litre$

= 0.7 moles

Substituting into equation (D.4)

$m = 0.7 \times 58.44$

= 40.908g NaCl is needed.

For 0.9M

Similarly, using equation (D.3) and (D.4)

$\mathrm{n} = 0.9\frac{\mathrm{mol}}{\mathrm{litre}} \times 1\mathrm{litre}$

= 0.9 moles

ISSN 2278–5469 EISSN 2278–5450 I OPEN ACCESS

Substituting into equation (D.4)
$m = 0.9 \times 58.44$
= 52.596g NaCl is needed.

For 1.0M:
Using equation (D.3) and D.4)
$n = 1.0 \frac{\text{mol}}{\text{litre}} \times 1\text{litre}$
= 1.0 moles
Substituting into equation (D.4)
$m = 1.0 \times 58.44$
= 58.44g NaCl is needed.

Data Sheet for Cassava

s/no	Preservative			STARCH - Casava (CV)			
				CV1		CV2	
	Type		Concentration (M)	ID	Viscosity (kg/ms) or m^2/s	ID	Viscosity (kg/ms) or m^2/s
1	Acid	H_2SO_4	0.1	CV1Ap1-0.1		CV2Ap1-0.1	
2			0.3	CV1Ap1-0.3		CV2Ap1-0.3	
3			0.5	CV1Ap1-0.5		CV2Ap1-0.5	
4			0.7	CV1Ap1-0.7		CV2Ap1-0.7	
5			0.9	CV1Ap1-0.9		CV2Ap1-0.9	
6			1	CV1Ap1-1.0		CV2Ap1-1.0	
7	Salt	NaCl	0.1	CV1Sp1-0.1		CV2Sp1-0.1	
8			0.3	CV1Sp1-0.3		CV2Sp1-0.3	
9			0.5	CV1Sp1-0.5		CV2Sp1-0.5	
10			0.7	CV1Sp1-0.7		CV2Sp1-0.7	
11			0.9	CV1Sp1-0.9		CV2Sp1-0.9	
12			1	CV1Sp1-1.0		CV2Sp1-1.0	
13	Neutral	Distilled H_2O		CV1-water		CV2-water	

Table for Viscosity Predictive Models for Casava Starch Specie CV1

Month	Mathematical model, Viscosity (y_0, y_1, y_2 and y_3); Concentration (x)	Error based on zero conc.(x=0) prediction		Error based on 1.0M conc.(x=1.0) prediction	
		Measured/Predicted	% Error	Measured/Predicted	% Error
CV1 in H_2SO_4					
0	$y_0 = -6735.1x^5 + 21746x^4 - 24086x^3 + 11703x^2 - 2312.2x + \mathbf{315.79}$ $R^2 = 1$	315.9/**315.79**	12.13	631.8/631.4	-0.06
1	$y_1 = -7145.8x^5 + 22491x^4 - 24261x^3 + 11304x^2 - 2132.2x +$	315.7/**316.59**	-0.24	571.9/572.59	0.12

ISSN 2278–5469 EISSN 2278–5450 I OPEN ACCESS

	316.59 $R^2 = 0.9993$				
2	$y_2 = -7136.1x^5 + 21762x^4 - 22913x^3 + 10420x^2 - 1936.1x +$ **341.54** $R^2 = 0.9999$	341.8/**341.54**	7.34	538.9/538.34	-0.10
3	$y_3 = -11201x^5 + 32474x^4 - 32243x^3 + 13319x^2 - 2130.7x +$ **367.1** $R^2 = 0.9996$	367.7/**367.1**	15.55	585.2/585.4	0.03
CV1 in NaCl					
0	$y_0 = -13049x^5 + 28518x^4 - 11795x^3 - 7824.9x^2 + 4529.7x +$ **318.86** $R^2 = 0.997$	315.9/**318.86**	11.60	652.9/697.66	6.85
1	$y_1 = 5319.7x^5 - 17921x^4 + 26554x^3 - 18152x^2 + 4493.4x +$ **314.64** $R^2 = 0.9992$	315.7/**314.64**	-9.01	591.1/608.74	2.98
2	$y_2 = 23600x^5 - 65653x^4 + 68052x^3 - 30832x^2 + 5044.1x +$ **332.94** $R^2 = 0.9192$	341.8/**332.94**	-4.67	557.21/544.04	-2.36
3	$y_3 = 15166x^5 - 33422x^4 + 23182x^3 - 3655.9x^2 - 1114.4x +$ **363.71** $R^2 = 0.9869$	367.7/**363.71**	10.46	553.01/519.41	-6.08

Table for Viscosity Predictive Models for Casava Starch Specie CV2

Month	Mathematical model, Viscosity (y_0, y_1, y_2 and y_3); Concentration (x)	Error based on zero conc.(x=0) prediction		Error based on 1.0M conc.(x=1.0) prediction	
		Measured/Predicted	% Error	Measured/Predicted	% Error
CV2 in H_2SO_4					
0	$y_0 = -6018.2x^5 + 22036x^4 - 26025x^3 + 12698x^2 - 2377.7x +$ **337.67** $R^2 = 0.9955$	340.3/**337.67**	-11.15	652.9/650.77	−0.33%
1	$y_1 = -5976.3x^5 + 20987x^4 - 24157x^3 + 11582x^2 - 2173.9x +$ **327.58** $R^2 = 0.9995$	328.3/**327.58**	-8.36	591.1/589.38	−0.29
2	$y_2 = -6384.4x^5 + 21040x^4 - 23510x^3 + 11249x^2 - 2195.5x +$ **356.94** ($R^2 = 0.998$)	358.3/**356.94**	-5.05	557.21/556.04	−0.21
3	$y_3 = -11146x^5 + 32006x^4 -$	377.6/**375.7**	-2.17	553.01/552.1	-0.16

ISSN 2278–5469 EISSN 2278–5450 I OPEN ACCESS

	$32119x^3 + 13880x^2 - 2444.6x +$ **375.7** ($R^2 = 0.9958$)				
CV2 in NaCl					
0	$y_0 = -13190x^5 + 32066x^4 - 18596x^3 - 3431.4x^2 + 3563.8x +$ **341.91** ($R^2 = 0.9991$)	340.3/**341.91**	-14.37	752.45/754.31	0.25
1	$y_1 = 6821x^5 - 18638x^4 + 23722x^3 - 15486x^2 + 3923.4x +$ **329.91** $R^2 = 0.9982$	328.3/**329.91**	-8.92	670.5/672.31	0.27
2	$y_2 = 26244x^5 - 68952x^4 + 67102x^3 - 28747x^2 + 4615.8x +$ **355.88** ($R^2 = 0.9922$)	358.3/**355.88**	-11.01	630.1/618.68	-1.81
3	$y_3 = 14033x^5 - 30075x^4 + 20088x^3 - 2804.8x^2 - 1066.4x +$ **373.** ($R^2 = 0.9878$)	377.6/**373.9**	2.93	552.47/548.7	-0.68

Funding

This study has not received any external funding.

Declaration of conflicting interests

The authors declare that there are no conflicts of interests.

Data and materials availability

All data associated with this study are present in the paper.

REFERENCES AND NOTES

1. Amani, G. G., Kamenan, A., Rolland-Sabaté, A. & Colonna. 2004. Stability of Yam Starch Gels During Processing, *African Journal of Biotechnology*, 4 (1), 94-101.
2. Akhabue, C. E., Ebewele, R.O. & Oyedoh E.A. (2009).Effects of Preservative Agents on Cassava Starch Adhesives, *Advanced Materials Research*. 10(5), 62-64.
3. Mooney, B. P. (2009). The Second Green Revolution? Production of Plant-based Biodegradable Plastics. *Biochemical Journal*, 418(2), 219-232.
4. Bajpai, P. (2018), Corrugated Containers, *Biermann's Handbook of Pulp and Paper* 2(3), 273-281.
5. Brunnschweiler, J., Luethi, D., Handschin, S., Farah, Z., Escher, F., & Conde-Petit, B. (2005). Isolation, Physicochemical Characterization and Application of Yam (Dioscorea spp.) Starch as Thickening and Gelling Agent. *Starch-Stärke*, *57*(3-4), 107-117.
6. Neelam K., Vijay S. & Lalit S. (2012), Various techniques for the modification of starch and the applications of its derivatives. *International research journal of pharmacy*, *3*(5), 25-31.
7. Kushairi, M. S., Rokiah, H., Othman N. S., Salim H., Wan, N. A., Norani, A. K., Nadiah, J & Lily Z. P. A. (2015), Evaluation of properties of starch-based adhesives and particleboard manufactured from them, *Journal of Adhesion Science and Technology*, 29(4)319-336.
8. Karama, L. B.,Grossmanna, M. V. E., Rui, S.S.F.S., Cristina, F.& Zaritzky, N. E.(2005), Gel Textural Characteristics of Corn, Cassava and Yam Starch Blends, *J. Starch/Starke.*57, 62–70.
9. Peter O. O., Ajisegiri, E. S. A, IDAH P. A. (2007) 'Production of Adhesives from Cassava'*Leonardo Electronic Journal of Practices and Technologies.* 10, 93-98.

10. Uchendu, N. O., Eze, S.O.O., Ugwu, O. P.C.,Enechi, O.C. &Udeh, S. M.C. (2013), Characterization of Different Varieties of Cassava Starch for Industrial Utilization. *IJRRPAS*. 3(3), 370-386.
11. Ukpaka, C.P., Amadi, S.A. & Gumus, R.H. (2009), Experimental Analysis of the Effect of Preservative Methods of Cassava Starch for Adhesives. *Global Journal of Engineering and Technology*. 2, 623-634.
12. Anahita N., Jim W. & Marc A. D.(2020). Using Lignin to Modify Starch-Based Adhesive Performance. *Journal of Chemical Engineering*, 4(3), 1-11.
13. Lee J., Park J. & Lee S., (2006), Effect of Starch and Base Paper Properties an Adhesion Problem of Corrugated Board, *Journal of Korea Technical Association of the Pulp and Paper Industry*. (38)

ISSN 2278–5469 EISSN 2278–5450 I OPEN ACCESS